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The contrast factors of dislocations in cubic crystals: the dislocation model of strain anisotropy in practice

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Abstract

It has been shown recently that in many cases strain anisotropy in powder diffraction can be well accounted for by the dislocation model of the mean square strain. The practical application assumes knowledge of the individual contrast factors C of dislocations related to particular Burgers, line and diffraction vectors or to the average contrast factors $\bar{C}$. A simple procedure for the experimental determination of $\bar{C}$ has been worked out, enabling the determination of the character of the dislocations in terms of a simple parameter q. The values of the individual C factors were determined numerically for a wide range of elastic constants for cubic crystals. The $\bar{C}$ factors and q parameters were parametrized by simple analytical functions, which can be used in a straightforward manner in numerical analyses, as *e.g.* in Rietveld structure refinement procedures.

1. Introduction

Anisotropic X-ray line broadening means that neither the full width at half-maximum (FWHM) nor the integral breadth nor the Fourier coefficients of diffraction profiles are monotonous functions of the diffraction vector or its square, $\mathbf{g}$ or $\mathbf{g}^2$, respectively (Williamson & Smallman, 1955; Caglioti *et al.*, 1958; Warren & Averbach, 1952; Warren, 1959). For the purpose of Rietveld structure refinement, different phenomenological solutions have been suggested to handle this problem (Suortti, 1993; Latroche *et al.*, 1995; Stephens, 1999; Scardi, 1998; Popa, 1998). Recently it has been shown that the dislocation model of the mean square strain, $\langle\varepsilon^2_{\mathbf{g},L}\rangle$ [where L is the Fourier length (Warren, 1959) and $\varepsilon_{\mathbf{g}}$ is the distortion tensor component in the $\mathbf{g}$ direction], can well account in many cases for anisotropic line broadening (Klimanek & Kuzel, 1988; Ungár & Borbély, 1996; Révész *et al.*, 1996; Ungár *et al.*, 1998, 1999; Ungár & Tichy, 1999; Wu *et al.*, 1998). Several attempts are also in progress to incorporate the dislocation model of strain anisotropy into the Rietveld structure refinement procedure (Ungár *et al.*, 1999; Wu *et al.*, 1998; Levine & Thomson, 1997). The dislocation model of $\langle\varepsilon^2_{\mathbf{g},L}\rangle$ is based on the contrast of dislocations varying with the relative orientation of the Burgers and line vectors of dislocations and the diffraction vector, $\mathbf{b}$, $\mathbf{l}$ and $\mathbf{g}$, respectively, in a similar way as in transmission electron microscopy (TEM) (Hirsch *et al.*, 1965). The contrast effect of dislocations on X-ray line broadening in cubic crystals assuming elastic isotropy was first evaluated by Krivoglaz (1969). In elastically anisotropic copper, Wilkens calculated the dislocation contrast factors in the late 1970s. The results were used and referenced in a number of papers as 'M. Wilkens, unpublished results' (Wilkens *et al.*, 1980; Ungár *et al.*, 1984, 1986). It has also been shown that the anisotropy of the elastic constants can have a strong effect on the contrast factors (see, for example, Table 3 of Wilkens *et al.*, 1980). A more comprehensive compilation of the dislocation contrast factors for copper taking into account elastic anisotropy has been presented by Wilkens (1987). A general formalism for the calculation of the contrast factors, especially for hexagonal crystals, has been given by Klimanek and Kuzel (Klimanek & Kuzel, 1988, 1989; Kuzel & Klimanek, 1988).

Since the dislocation contrast factors depend on the different possible combinations of $\mathbf{b}$, $\mathbf{l}$ and $\mathbf{g}$ and on the anisotropic elastic constants, these being a large variety of variables, it is not easy to find a concise representation of these factors. One aim of this paper is to present average contrast factors in a more-or-less general and concise form. In the case of a particular crystal system, the combinations of $\mathbf{b}$, $\mathbf{l}$ and $\mathbf{g}$ depend on the possible slip systems and their population by dislocations, which makes the evaluation of $\langle\varepsilon^2_{\mathbf{g},L}\rangle$ in a general case rather difficult (Stephens, 1999; Ungár & Borbély, 1996). However, in the case of untextured polycrystals or if the different slip systems are populated randomly by dislocations, the contrast factors can be averaged and the evaluation procedure becomes simpler and more straightforward (Ungár & Tichy, 1999). In the present work, both individual and averaged contrast factors are compiled as functions of the elastic constants for the possible dislocations in face-centred cubic (f.c.c.) and body-centred cubic (b.c.c.) crystals. The average contrast factors are parametrized by simple analytical functions, which can be easily incorporated in computational work. It is shown that the compiled average contrast factors

can be used as a simple tool for the analysis of microstructure in terms of coherent domain size, dislocation density and dislocation character in the f.c.c. and b.c.c. crystal systems.

2. The contrast factors of dislocations

The Fourier coefficients of diffraction profiles can be given as the product of the 'size' and 'distortion' coefficients, A_L^S and A_L^D (Warren, 1959):

$$A_L = A_L^S A_L^D. \tag{1}$$

L is the Fourier length, defined as $L = na_3$, where n are integers and a_3 is the unit of the Fourier length in the direction of the diffraction vector $\mathbf{g}$:

$$a_3 = \lambda/[2(\sin\theta_2 - \sin\theta_1)], \tag{2}$$

where the line profile is measured from θ_1 to θ_2 and λ is the wavelength of the X-rays. Warren (1959) has shown that the Fourier coefficients can be written as

$$\ln A_L(\mathbf{g}) \simeq \ln A_L^S - 2\pi^2 L^2 g^2 \langle \varepsilon_{\mathbf{g}}^2 \rangle, \tag{3}$$

where $\langle \varepsilon_{\mathbf{g}}^2 \rangle$ is the mean square strain in the direction of the diffraction vector. It can be evaluated for different kinds of lattice defects, especially for dislocations (Wilkens, 1970; Ungár *et al.*, 1989; Groma *et al.*, 1988; Krivoglaz, 1996):

$$\langle \varepsilon_{\mathbf{g},L}^2 \rangle \simeq (\rho C b^2/4\pi) \ln(R_e/L), \tag{4}$$

where ρ is the density, R_e the effective outer cut-off radius, b the absolute value of the Burgers vector and C the contrast factor of dislocations. Equation (4) shows that for dislocations, $\langle \varepsilon_{\mathbf{g},L}^2 \rangle$ is dependent on L, as observed by experiment (Warren, 1959). The contrast factors depend on the relative orientations of the line and Burgers vectors of dislocations and the diffraction vector. Consider a straight dislocation parallel to the z axis of an x, y, z Cartesian coordinate system. The displacement field of such a dislocation can be given by the polar coordinates (j, r) in the xy plane. With this notation, the contrast factor C can be given as (Wilkens, 1970; Klimanek & Kuzel, 1988; Groma *et al.*, 1988)

$$C = (1/\pi) \int_0^{2\pi} \mathrm{d}\varphi \, K^2(\varphi), \tag{5a}$$

$$K(\varphi) = \sum_{i=1}^{3} \sum_{j=1}^{2} \gamma_i \gamma_j \beta_{ij}(\varphi), \tag{5b}$$

$$\beta_{ij} = \frac{2\pi r}{b} \frac{\partial \mathbf{u}_i}{\partial \mathbf{x}_j}, \quad i = 1, 2, 3 \quad \text{and} \quad J = 1, 2, \tag{5c}$$

where $K(\varphi)$ is a trigonometric polynomial, γ_i and γ_j are the direction cosines of the diffraction vector in the xy plane, $\partial \mathbf{u}_i/\partial \mathbf{x}_j$ is the distortion tensor, $\mathbf{u}_i$ is the displacement field of the dislocation and r is the length variable in the polar coordinate system. Equations (5) can be evaluated numerically by using the elastic constants of a crystal. Because of the linear superposition of the displacement fields of dislocations, the contrast factors corresponding to a dislocation system can be obtained by the linear superposition of the individual contrast factors as weighted averages (Wilkens, 1970).

3. The average contrast factors of dislocations in cubic crystals

In a recent paper, it has been shown that in the cubic crystal system the average contrast factors are a linear function of the fourth-order invariant of the *hkl* indices of the different reflections (Ungár & Tichy, 1999):

$$\bar{C} = A - B(h^2k^2 + h^2l^2 + k^2l^2)/(h^2 + k^2 + l^2)^2, \tag{6}$$

where A and B are constants depending on the elastic constants of the crystal. The value of A is the average contrast factor corresponding to the $h00$ reflection: $\bar{C}_{h00} = A$. Denoting the fourth-order ratio in the above equation by H^2, equation (6) can be written as

$$\bar{C} = \bar{C}_{h00}(1 - qH^2), \tag{7}$$

where $q = B/A$. This equation shows that in the case of untextured polycrystals or randomly populated Burgers vectors, the average contrast factors can be obtained if $\bar{C}_{h00}$ and q are known.

4. The values of the contrast factors in cubic materials

When the material has a pronounced texture with particular slip systems populated by dislocations in the different texture components, the average dislocation contrast factors cannot be used. In these cases the individual contrast factors are needed in order to evaluate strain anisotropy. On the other hand, the average C factor values, $\bar{C}$, were calculated from these individual contrast factors. For these reasons, before presenting the average C factors, representative values of the individual contrast factors are given in Tables 1–4, for $\mathbf{b} = a/2\langle 110 \rangle$ and $\mathbf{b} = a/2\langle 111 \rangle$ type dislocations in the $\{111\}\langle 110 \rangle$ and $\{110\}\langle 111 \rangle$ slip systems, respectively. The individual C factors were calculated for the 111, 200 and 220 reflections in the f.c.c. system and for the 110, 200 and 222 reflections in the b.c.c. system, in each case for pure edge and pure screw dislocations separately. It follows from the structure of equation (5) that the values of C depend only on the ratios of the elastic constants. This means that the three elastic constants, c_{11}, c_{12} and c_{44}, in the cubic system can be reduced to two parameters. We select the elastic anisotropy $A_i = 2c_{44}/(c_{11} - c_{12})$ and the ratio c_{12}/c_{44}. All individual C factors were determined as a function of A_i and c_{12}/c_{44} in the ranges 0.5–8 and 0.5–3, respectively, since these are the ranges in which the elastic constants of most materials vary (Hearmon, 1966). The average contrast factors can be obtained as weighted averages of the individual contrast

Table 1. *Representative values of the contrast factors for screw dislocations in f.c.c. crystals as a function of the elastic anisotropy A_i*

In the present case the values are independent of c_{12}/c_{44}.

hkl	*m*	*j*	$A_i = 0.5$	$A_i = 0.75$	$A_i = 1$	$A_i = 1.5$	$A_i = 2$	$A_i = 4$	$A_i = 8$
200	2	1	0.1767	0.2165	0.2500	0.3062	0.3536	0.5000	0.7071
	1	2	0	0	0	0	0	0	0
220	2	2	0.2210	0.1985	0.1875	0.1786	0.1768	0.1875	0.2210
	1	1	0	0	0	0	0	0	0
111	1	1	0.3143	0.2566	0.2222	0.1814	0.1571	0.1111	0.0786
	1	2	0	0	0	0	0	0	0

The combinations of Burgers and line vectors for the *C* factor values

j	**b**	**l**
1	−1−10	−1−10
2	01−1	01−1

factors as mentioned at the end of §2 (Wilkens, 1970). The weight factor is the multiplicity of the slip systems providing the same *C* value at a particular *hkl* value and is denoted by *m* in Tables 1–4. We note that the averaging could also be performed by fixing a particular slip system and varying the planes for given *hkl*. Here we have selected averaging over the slip systems since in this case the *C* values corresponding to active slip systems can be selected for the evaluation of strain anisotropy. The different slip systems giving different *C* values are listed in Tables 1–4, where the slip systems are denoted by *j*.

5. The values of $\bar{C}_{h00}$ in f.c.c. and b.c.c. crystals

The average values of the contrast factors were calculated for the most common slip systems in the f.c.c. and b.c.c. systems (Honeycombe, 1984): ⟨110⟩{111} and ⟨111⟩{110}, respectively. Equation (7) shows that the two parameters for the practical determination of the average *C* factors as a function of H^2 are $\bar{C}_{h00}$ and *q*. The $\bar{C}_{h00}$ values for edge and screw dislocations were obtained by arithmetic averages of the individual C_{h00} values and are shown as functions of A_i and c_{12}/c_{44} in Figs. 1 and 2. In the case of f.c.c. crystals, no dependence on c_{12}/c_{44} has been found for screw dislocations. For edge dislocations, a relatively strong dependence on c_{12}/c_{44} can be observed. The A_i dependence of $\bar{C}_{h00}$ at different c_{12}/c_{44} values can be parametrized by the following function:

$$\bar{C}_{h00} = a[1 - \exp(-A_i/b)] + cA_i + d. \qquad (8)$$

In the case of screw dislocations in f.c.c. crystals, a = 0.1740, b = 1.9522, c = 0.0293 and d = 0.0662, independent of c_{12}/c_{44}. For edge dislocations in f.c.c. crystals, the values of *a* to *d* are listed in Table 5. In the case of screw dislocations in b.c.c. crystals when $c_{12}/c_{44} = 1$, $a = 0.1740$, $b = 1.9522$, $c = 0.0293$ and $d = 0.0662$. For $c_{12}/c_{44} = 0.5$ and 2, the parameter values are only slightly different, as can be seen in Fig. 1(*c*). For edge dislocations in b.c.c. crystals, the values of *a* to *d* are listed in Table 6.

6. The values of *q* in f.c.c. and b.c.c. crystals

The average values of the contrast factors, $\bar{C}(A_i, c_{12}/c_{44})$, for the 111, 200 and 220 reflections for pure edge and pure screw dislocations in f.c.c. crystals with the $a/2\langle 110\rangle\{111\}$ slip system can be obtained from Tables 1 and 2. The values of *q* were obtained from these contrast factors by using equation (7). Fig. 2(*a*) shows *q* in f.c.c. crystals as a function of A_i for pure screw and pure edge dislocations, the latter when $c_{12}/c_{44} = 1$. For screw dislocations, *q* turns out to be independent of c_{12}/c_{44} in the rage between 0.5 and 3 (at least in f.c.c. crystals). For edge dislocations, *q* reveals a weak dependence on c_{12}/c_{44} from 0.5 to 1, whereas in the range between 1 and 3 this dependence becomes marginal. The difference in *q* for edge and screw dislocations is, however, substantial, as can be seen in Fig. 2(*a*).

The A_i dependence of *q* at different c_{12}/c_{44} values can be parametrized by the same kind of equation as (8):

$$q = a[1 - \exp(-A_i/b)] + cA_i + d. \qquad (9)$$

In the case of screw dislocations in f.c.c. crystals, a = 5.4252, b = 0.7196, c = 0.0690 and d = −3.1970, independent of the c_{12}/c_{44} values. For the case of edge dislocations, the values of *a* to *d* are listed in Table 7.

The *q* values in b.c.c. crystals are shown in Fig. 2(*b*). A relatively large difference can be observed, especially in the range of A_i between 0.5 and 4. In the case of screw dislocations, a weak dependence on c_{12}/c_{44} has been found, as indicated by the slightly varying parameter values in Table 8. In the case of edge dislocations, the dependence on c_{12}/c_{44} is stronger, as can be seen in Fig. 2(*c*) and by the markedly changing parameter values in Table 9.

Table 2. *Representative values of the contrast factors for edge dislocations in f.c.c. crystals as a function of the elastic anisotropy A_i and the ratio c_{12}/c_{44}*

hkl	*m*	*j*	$A_i = 0.5$	$A_i = 0.75$	$A_i = 0.98$	$A_i = 1.5$	$A_i = 2$	$A_i = 4$	$A_i = 8$
$c_{12}/c_{44} = 0.5$									
200	2	1	0.2099	0.2440	0.2676	0.3076	0.3369	0.4213	0.5322
	1	2	0.0071	0.0088	0.0109	0.0173	0.0245	0.0528	0.0932
220	2	1	0.1676	0.1836	0.1933	0.2063	0.2132	0.2260	0.2392
	2	4	0.0418	0.0379	0.0363	0.0353	0.0358	0.0417	0.0561
	1	1	0.6406	0.6029	0.5814	0.5544	0.5391	0.5102	0.4927
	1	3	0.1150	0.0660	0.0456	0.0294	0.0246	0.0464	0.0867
111	2	1	0.3427	0.3222	0.3103	0.2922	0.2801	0.2515	0.2258
	1	3	0.1595	0.1190	0.1011	0.0817	0.0721	0.0535	0.0382
	1	4	0.0094	0.0034	0.0013	0.0013	0.0036	0.0161	0.0357
$c_{12}/c_{44} = 1$									
200	2	1	0.2028	0.2362	0.2604	0.3039	0.3378	0.4383	0.5622
	1	2	0.0059	0.0086	0.0120	0.0215	0.0313	0.0669	0.1133
220	2	1	0.1671	0.1833	0.1944	0.2120	0.2239	0.2528	0.2823
	2	4	0.0416	0.0369	0.0348	0.0335	0.0342	0.0424	0.0596
	1	1	0.6305	0.5838	0.5574	0.5208	0.4994	0.4585	0.4332
	1	3	0.1327	0.0773	0.0511	0.0237	0.0140	0.0153	0.0414
111	2	1	0.3372	0.3130	0.2987	0.0005	0.2638	0.2338	0.2096
	1	3	0.1816	0.1352	0.1126	0.0773	0.0712	0.0458	0.0294
	1	4	0.0106	0.0041	0.0015	0.0223	0.0022	0.0132	0.0311
$c_{12}/c_{44} = 2$									
200	2	1	0.1981	0.2345	0.2625	0.3152	0.3572	0.4778	0.6119
	1	2	0.0069	0.0117	0.0169	0.0299	0.0425	0.0849	0.1367
220	2	1	0.1793	0.2005	0.2160	0.2425	0.2612	0.3066	0.3490
	2	4	0.0417	0.0359	0.0333	0.0320	0.0332	0.0443	0.0638
	1	1	0.6198	0.5650	0.5335	0.4897	0.4644	0.4174	0.3891
	1	3	0.1643	0.1028	0.0715	0.0357	0.0208	0.0119	0.0298
111	2	1	0.3339	0.3064	0.2901	0.2667	0.2523	0.2227	0.2012
	1	3	0.2303	0.1823	0.1579	0.1278	0.1122	0.0855	0.0700
	1	4	0.0124	0.0052	0.0021	0.0004	0.0016	0.0120	0.0292
$c_{12}/c_{44} = 3$									
200	2	1	0.1985	0.2384	0.2697	0.3290	0.3758	0.5060	0.6427
	1	2	0.0094	0.0155	0.0217	0.0364	0.0502	0.0958	0.1499
220	2	1	0.1958	0.2213	0.2399	0.2714	0.2932	0.3452	0.3920
	2	4	0.0420	0.0354	0.0327	0.0314	0.0330	0.0455	0.0659
	1	1	0.6148	0.5562	0.5224	0.4758	0.4491	0.4003	0.3709
	1	3	0.1895	0.1248	0.0911	0.0518	0.0348	0.0220	0.0362
111	2	1	0.2741	0.2264	0.2017	0.1708	0.1545	0.1265	0.1105
	1	3	0.3346	0.3053	0.2882	0.2636	0.2489	0.2195	0.1992
	1	4	0.0137	0.0060	0.0027	0.0006	0.0017	0.0121	0.0291

The combinations of Burgers and line vectors for the *C* factor values

j	**b**	**l**
1	−10−1	−121
2	0−1−1	−21−1
3	−110	−1−12
4	−101	121

7. The practical or experimental determination of the average contrast factors

The *classical* Williamson–Hall (Williamson & Hall, 1953) plot can be solved for dislocated crystals and has been called the *modified* Williamson–Hall plot (Ungár & Borbély, 1996):

$$\Delta K \simeq 0.9/D + (\pi M^2 b^2/2)^{1/2} \rho^{1/2} K C^{1/2} + O(K^2 C). \tag{10}$$

In its quadratic form it is (Williamson & Hall, 1953)

$$(\Delta K)^2 \simeq (0.9/D)^2 + (\pi M^2 b^2/2) \rho K^2 C \pm O(K^4 C^2). \tag{11}$$

Table 3. *Representative values of contrast factors for screw dislocations in b.c.c. crystals as a function of the elastic anisotropy A_i and the ratio c_{12}/c_{44}*

hkl	*m*	*j*	$A_i = 0.5$	$A_i = 0.75$	$A_i = 1$	$A_i = 1.5$	$A_i = 2$	$A_i = 3$	$A_i = 4$	$A_i = 8$
$c_{12}/c_{44} = 0.5$										
110	1	1	0.2638	0.2381	0.2222	0.2032	0.1921	0.1797	0.1730	0.1638
	1	2	0.0227	0.0037	0	0.0059	0.0154	0.0323	0.0450	0.0745
200	1	1	0.1545	0.1928	0.2222	02642	0.2930	0.3309	0.3560	0.4161
222	3	2	0.1860	0.1292	0.0988	0.0685	0.0542	0.0414	0.0356	0.0269
	1	1	0	0	0	0	0	0	0	0
$c_{12}/c_{44} = 1$										
110	1	1	0.2624	0.2374	0.2222	0.2041	0.1937	0.1819	0.1755	0.1673
	1	2	0.0212	0.0033	0	0.0053	0.0136	0.0285	0.0401	0.0688
200	1	1	0.1563	0.1940	02222	0.2621	0.2888	0.3246	0.3493	0.4129
222	3	2	0.1827	0.1278	0.0988	0.0696	0.0559	0.0428	0.0364	0.0263
	1	1	0	0	0	0	0	0	0	0
$c_{12}/c_{44} = 2$										
110	1	1	0.2606	0.2366	0.2222	0.2053	0.1955	0.1844	0.1784	0.1712
	1	2	0.0195	0.0030	0	0.0046	0.0120	0.0257	0.0365	0.0653
200	1	1	0.1590	0.1956	0.2222	0.2591	0.2842	0.3186	0.3431	0.4113
222	3	2	0.1782	0.1260	0.0988	0.0715	0.0582	0.0451	0.0385	0.0274
	1	1	0	0	0	0	0	0	0	0

The combinations of Burgers and line vectors for the *C* factor values

j	**b**	**l**
1	111	111
2	1−11	1−11

where D is the average particle size, M is a constant depending on the effective outer cut-off radius of dislocations, $K = 2\sin\theta/\lambda$, ΔK is the FWHM and O indicates non-interpreted higher-order terms. Inserting equation (7) into (11) yields

$$[(\Delta K)^2 - \alpha]/K^2 \simeq \beta\bar{C}_{h00}(1 - qH^2), \qquad (12)$$

where $\alpha = (0.9/D)^2$ and $\beta = \pi M^2 b^2 \rho/2$. From the linear regression of the left-hand side of equation (12) *versus* H^2, the parameter q can be determined experimentally. The successful operation of equation (12) has been shown for submicrometre-grain-size copper, ball-milled iron and Rb_3C_{60} powder by Ungár & Tichy (1999) and a cubic Li–Mn spinel by Ungár *et al.* (1999). The FWHM can be corrected for stacking faults by the method of Warren (1959, p. 177), which has been adapted for the present evaluation procedure according to equations (14) and (16) of Ungár *et al.* (1998). Accordingly, equation (12) becomes

$$\{[\Delta K - \beta' W(g)]^2 - \alpha\}/K^2 \simeq \beta\bar{C}_{h00}(1 - qH^2), \qquad (13)$$

where $W(g) = 0.43, 1.0, 0.71$ and 0.45 for the 111, 200, 220 and 311 type reflections, respectively, and β' is a fitting parameter related to the stacking-fault probability.

Here we note that the experimental values of q can also be obtained by inserting (7) into (10) and solving it by the least-squares method. Experience has shown that the q values obtained by the two different procedures are equal within experimental error.

8. The experimental determination of q in three different examples

8.1. *The experimental determination of q in a severely deformed submicrometre-grain-size copper specimen*

Polycrystalline 99.98% copper of 300 μm initial grain size was deformed by the method of equal-channel angular pressing (ECA), producing submicrometre grain sizes in the range 50–500 nm (Valiev *et al.*, 1994). Line profiles of the first six reflections were measured by a special high-resolution double-crystal diffractometer, with negligible instrumental line broadening (Ungár & Borbély, 1996). The classical Williamson–Hall plot of the FWHM has revealed a strong strain anisotropy, which can be seen in Fig. 1 of Ungár & Borbély (1996). The plot of the FWHM according to equation (12) is shown in Fig. 3(*a*) herein. The best linear regression is obtained with the value of $a = 6 \times 10^{-6}$ (nm^{-2}), providing $q = 2.15$ (2). The elastic constants of Cu are $c_{11} = 166.1$, $c_{12} = 119.9$ and $c_{44} = 75.6$ GPa (Hearmon, 1966). With these values, $A_i = 3.21$ and $c_{12}/c_{44} = 1.608$. The value of $\bar{C}_{h00}$ can be obtained from the plots in Figs. 1(*a*) and 1(*b*): $\bar{C}_{h00} = 0.3040$, where it is assumed that edge and screw dislocations are present in equal proportion. With this, the average contrast factors for this specimen are

$$\bar{C} = 0.3040(1 - 2.01H^2). \qquad (14)$$

The modified Williamson–Hall plot with these contrast factors is shown in Fig. 3(*b*). It can be seen that the values of the FWHM are parabolic in $KC^{1/2}$. The inter-

Table 4. *Representative values of contrast factors for edge dislocations in b.c.c. crystals as a function of the elastic anisotropy A_i and the ratio c_{12}/c_{44}*

hkl	*m*	*j*	$A_i = 0.5$	$A_i = 0.75$	$A_i = 1$	$A_i = 1.5$	$A_i = 2$	$A_i = 3$	$A_i = 4$	$A_i = 8$
$c_{12}/c_{44} = 0.5$										
110	2	1	0.3553	0.3684	1.1968	0.3892	0.3974	0.4099	0.4208	0.4630
	1	2	0.3269	0.2850	0.8108	0.2214	0.1966	0.1634	0.1419	0.1027
	1	3	0.1379	0.1121	0.3573	0.0878	0.0803	0.0696	0.0617	0.0452
	2	4	0.0138	0.0079	0.0223	0.0075	0.0115	0.0229	0.0367	0.0974
200	2	1	0.1505	0.1793	0.6556	0.2431	0.2801	0.3518	0.4230	0.6968
	1	2	0.0579	0.0617	0.2027	0.0687	0.0724	0.0788	0.0843	0.1011
222	1	1	0.6545	0.6110	1.8244	0.5348	0.5011	0.4508	0.4136	0.3227
	2	3	0.1213	0.1075	0.3429	0.0968	0.0934	0.0871	0.0815	0.0656
	1	4	0.0128	0.0092	0.0225	0.0051	0.0041	0.0037	0.0043	0.0094
$c_{12}/c_{44} = 1$										
110	2	1	0.3466	0.3571	1.3182	0.3771	0.3870	0.4047	0.4212	0.4811
	1	2	0.3241	0.2776	0.8619	0.2061	0.1792	0.1454	0.1256	0.0945
	1	3	0.1599	0.1285	0.5809	0.0908	0.0784	0.6350	0.0548	0.0418
	2	4	0.0165	0.0092	0.0363	0.0086	0.0142	0.0308	0.0501	0.1280
200	2	1	0.1440	0.1737	0.7807	0.2522	0.3033	0.4043	0.5023	0.8492
	1	2	0.0551	0.0588	0.2155	0.0668	0.0714	0.0795	0.0865	0.1064
222	1	1	0.6394	0.5906	1.9392	0.5045	0.4670	0.4128	0.3747	0.2873
	2	3	0.1353	0.1160	0.4688	0.0932	0.0851	0.0739	0.0665	0.0127
	1	4	0.0132	0.0091	0.0239	0.0046	0.0038	0.0041	0.0055	0.0509
$c_{12}/c_{44} = 2$										
110	2	1	0.3419	0.3520	1.4939	0.3750	0.3878	0.4111	0.4323	0.5054
	1	2	0.3213	0.2699	0.9285	0.1926	0.1657	0.1343	0.1176	0.0942
	1	3	0.2074	0.1752	0.9433	0.1334	0.1196	0.1030	0.0934	0.0764
	2	4	0.0216	0.0126	0.0590	0.0125	0.0206	0.0401	0.0678	0.1600
200	2	1	0.1414	0.1773	0.9734	0.2835	0.3533	0.4865	06083	1.0094
	1	2	0.0521	0.0558	0.2321	0.0650	0.0705	0.0804	0.0886	0.1108
222	1	1	0.6222	0.5692	2.0892	0.4767	0.4376	0.3828	0.3435	0.2632
	2	3	0.1656	0.1415	0.6697	0.1084	0.0964	0.0812	0.0718	0.0543
	1	4	0.0138	0.0091	0.0258	0.0043	0.0039	0.0052	0.0074	0.0163

The combinations of Burgers and line vectors for the *C* factor values

j	**b**	**l**
1	111	2−1−1
2	111	11−2
3	1−11	−112
4	1−11	121

section at $K = 0$ gives $D = 350$ (10) nm for the average particle size corresponding to the FWHM, in good agreement with the evaluation performed earlier (Ungár & Borbély, 1996). The values of q corresponding to $A_i = 3.21$ and $c_{12}/c_{44} = 1.608$ can be obtained for pure screw or pure edge dislocations from the plots in Fig. 2(*a*) or, with a higher precision, by using equation (9) and the parameter values in Table 7: $q_{\rm screw} = 2.37$ and $q_{\rm edge} = 1.63$, respectively. The experimental value, $q_{\rm exp} = 2.01$, is exactly the arithmetic average of the two numerically calculated q values. The straightforward interpretation of this result is that the character of the prevailing dislocations is half edge, half screw.

8.2. *The experimental determination of q in an electrodeposited nanocrystalline nickel specimen*

A nanocrystalline Ni foil of diameter 50 mm and thickness 8 μm was prepared by pulse-plating electrodeposition from aqueous solution onto cold-rolled polycrystalline Cu substrates by Tóth-Kádár *et al.* (1996). The X-ray line profiles were measured using a special high-resolution double-crystal diffractometer, with negligible instrumental broadening (Ungár *et al.*, 1999). The classical Williamson–Hall plot of the FWHM values of the first six reflections shows a strong strain anisotropy, as can be seen in Fig. 4(*a*). The same values of the FWHM are plotted according to equation (11) in Fig. 4(*b*). The best values of α and q were $\alpha = 6.5\ (5) \times 10^{-4}$ nm^{-2} and $q_{\rm exp} = 2.35\ (2)$. The elastic constants of Ni are $c_{11} = 243.6$, $c_{12} = 149.4$ and $c_{44} = 119.6$ GPa (Hearmon, 1966). With these values, $A_i = 2.54$ and $c_{12}/c_{44} = 1.25$. The value of $\bar{C}_{h00}$ can be obtained from the plots in Figs. 1(*a*) and 1(*b*): $\bar{C}_{h00} = 0.266$, where it is assumed that edge and screw dislocations are present in equal proportion. With this, the average contrast factors for this specimen are

$$\bar{C} = 0.266(1 - 2.35H^2). \quad (15)$$

The modified Williamson—Hall plot with these contrast factors is shown in Fig. 4(*c*). It can be seen that the values of the FWHM are parabolic in $KC^{1/2}$. The intersection at $K = 0$ gives $D = 50$ (5) nm for the average particle size corresponding to the FWHM, in good agreement with the evaluation performed earlier (Ungár *et al.*, 1999). The values of q corresponding to $A_i = 2.54$ and $c_{12}/c_{44} = 1.25$ can be obtained for pure screw or pure edge dislocations in an f.c.c. crystal from the plots in Fig. 2(*a*) or, with a higher precision by using equation (9) and the parameter values in Table 7: $q_{\rm screw} = 2.23$ and $q_{\rm edge} = 1.38$, respectively. The experimental value, $q_{\rm exp} = 2.35$ (2), is out of this range, even considering its uncertainty. The discrepancy can be solved by finding a type of dislocation which is still compatible with the f.c.c. lattice but provides a larger range for the values of q. A look at Fig. 2(*b*) shows that the ⟨111⟩{110} b.c.c. type dislocations yield a higher range for the q values for the same elastic constants: $q_{\rm screw} = 2.63$ and $q_{\rm edge} = 1.37$. The ⟨111⟩{110} type dislocations in an f.c.c. lattice are prismatic loops, observed with considerable density in TEM micrographs of the present specimen (Tóth-Kádár *et al.*, 1996).

8.3. *The experimental determination of q in a Rb-doped Rb_3C_{60} fullerite powder specimen*

The FWHM parameters of the Rb-doped Rb_3C_{60} fullerite were measured at the high-resolution powder diffractometer beamline X3B1 of the National Synchrotron Light Source at Brookhaven National Laboratory by Fischer *et al.* (1995). The FWHM values of the physical profiles show a pronounced strain anisotropy, as can be seen in Fig. 2 of Fischer *et al.* (1995). The same data are plotted according to equation (12) in Fig. 5(*a*) herein. An excellent linear regression can be obtained with $\alpha \simeq 3 \times 10^{-4}$ (nm^{-2}). The value of $q_{\rm exp}$ is 2.47 (3). Since the elastic constants of this material are not known, it is not possible to give the exact value of $\bar{C}_{h00}$. However, Figs. 1(*a*)–1(*d*) suggest that a value around 0.3 is probable. With this, the average contrast factors for this material are

$$\bar{C} \simeq 0.3(1 - 2.47H^2). \quad (16)$$

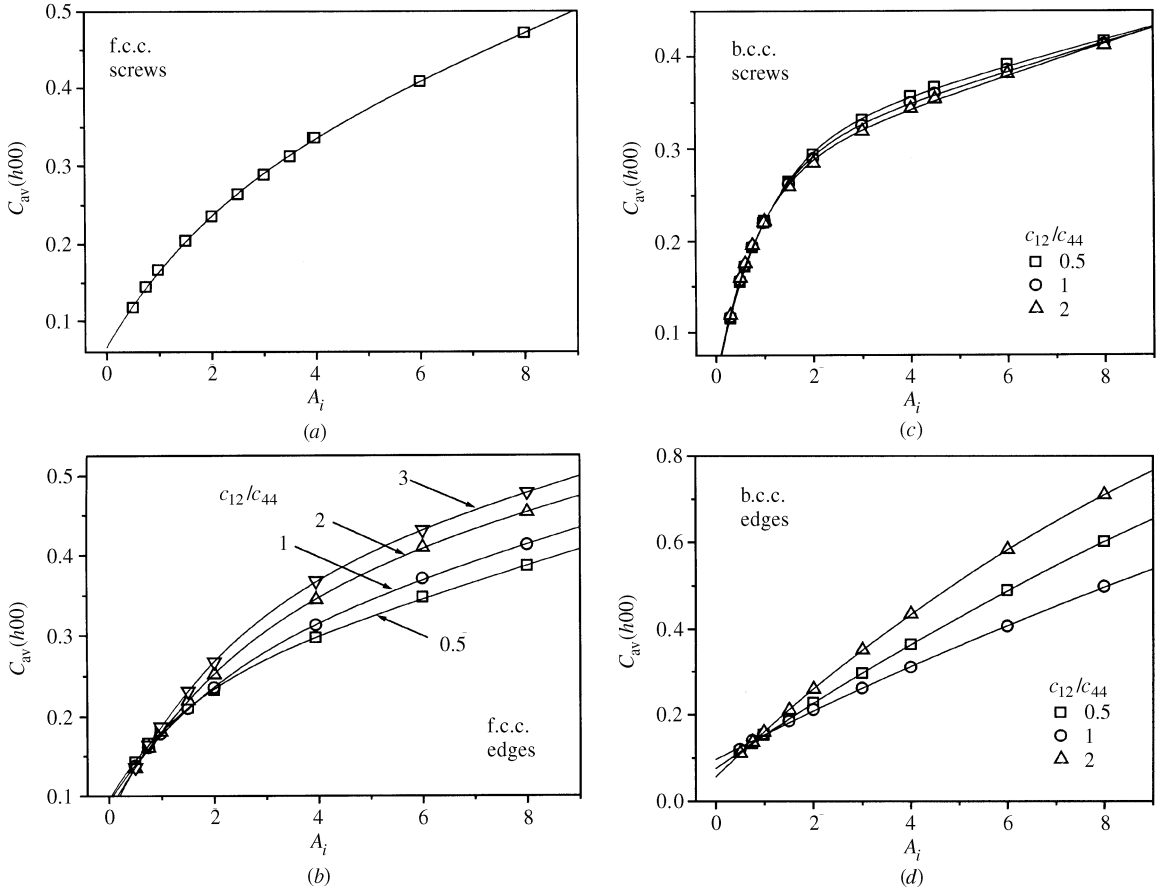

Fig. 1. The average C factors corresponding to the $h00$ reflections in the case of (*a*) f.c.c. screw, (*b*) f.c.c. edge, (*c*) b.c.c. screw and (*d*) b.c.c. edge dislocations as functions of the elastic anisotropy A_i and the ratio of the elastic constants c_{12}/c_{44}.

The modified Williamson–Hall plot with these contrast factors is shown in Fig. 5(*b*). It can be seen that the FWHM is almost linear. The intersection at $K = 0$ gives $D = 100\,(5)$ nm for the average particle size corresponding to the FWHM.

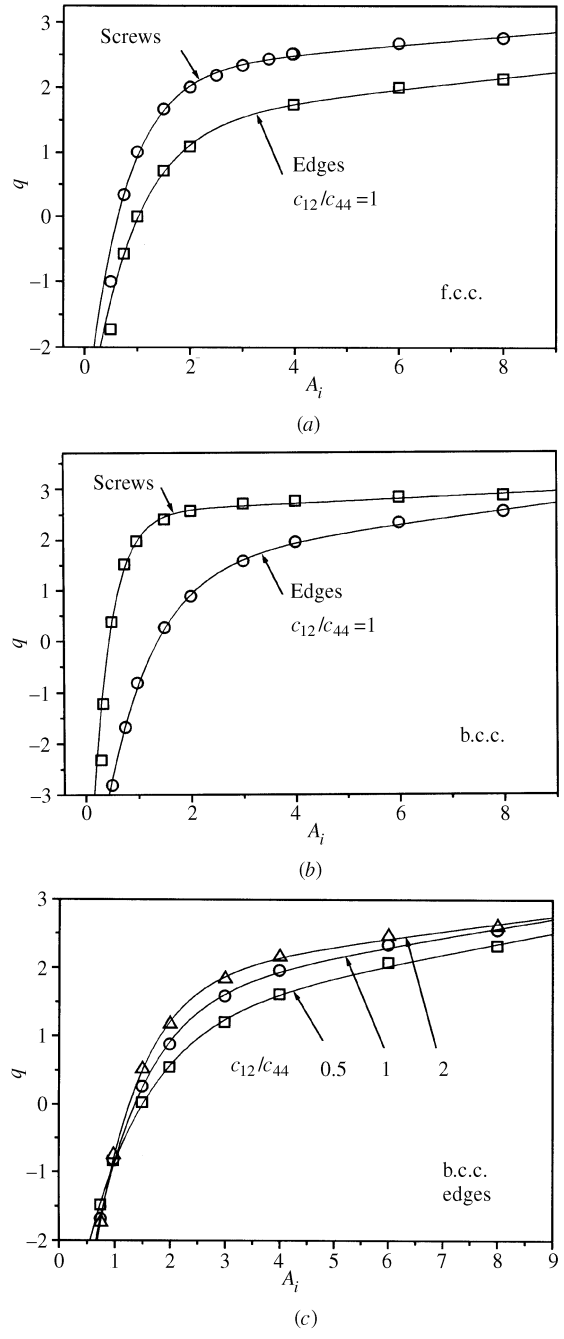

Fig. 2. The parameter q as a function of the elastic anisotropy A_i and the ratio of the elastic constants c_{12}/c_{44}: (*a*) in the case of f.c.c. screw and edge dislocations, the latter when $c_{12}/c_{44} = 1$; (*b*) in the case of b.c.c. screw and edge dislocations, the latter when $c_{12}/c_{44} = 1$; (*c*) in the case of b.c.c. edge dislocations at different c_{12}/c_{44} ratios.

Table 5. *The parameters a, b, c and d of equation (8) for $\bar{C}_{h00}$ in the case of edge dislocations for the f.c.c. crystal system*

	$c_{12}/c_{44} = 0.5$	$c_{12}/c_{44} = 1$	$c_{12}/c_{44} = 2$	$c_{12}/c_{44} = 3$
a	0.1312	0.1687	0.2438	0.2635
b	1.4284	2.0400	2.4243	2.1880
c	0.0201	0.0194	0.0172	0.0186
d	0.0954	0.0926	0.0816	0.0731

Table 6. *The parameters a, b, c and d of equation (8) for $\bar{C}_{h00}$ in the case of edge dislocations in the b.c.c. crystal system*

	$c_{12}/c_{44} = 0.5$	$c_{12}/c_{44} = 1$	$c_{12}/c_{44} = 2$
a	1.4948	1.6690	1.4023
b	25.671	21.124	12.739
c	0.0	0.0	0.0
d	0.0966	0.0757	0.0563

Table 7. *The parameters a, b, c and d of equation (9) for q in the case of edge dislocations in the f.c.c. crystal system*

	$c_{12}/c_{44} = 0.5$	$c_{12}/c_{44} = 1$	$c_{12}/c_{44} = 2$	$c_{12}/c_{44} = 3$
a	4.0327	4.8608	5.8282	6.3398
b	0.8846	0.8687	0.8098	0.7751
c	0.0986	0.0896	0.0828	0.0813
d	−2.8225	−3.4280	−4.297	−4.8129

Table 8. *The parameters a, b, c and d of equation (9) for q in the case of screw dislocations in the b.c.c. crystal system*

	$c_{12}/c_{44} = 0.5$	$c_{12}/c_{44} = 1$	$c_{12}/c_{44} = 2$
a	7.5149	8.6590	6.0725
b	0.3818	0.3730	0.4338
c	0.0478	0.0424	0.0415
d	−4.9826	−6.074	−3.5021

Table 9. *The parameters a, b, c and d of equation (9) for q in the case of edge dislocations in the b.c.c. crystal system*

	$c_{12}/c_{44} = 0.5$	$c_{12}/c_{44} = 1$	$c_{12}/c_{44} = 2$
a	5.3020	7.2361	8.8331
b	1.0945	0.9285	0.8241
c	0.1540	0.1359	0.1078
d	−4.1841	−5.7484	−7.0570

The experimental value of $q_{\rm exp} = 2.47\,(3)$ may be discussed as follows. Assuming half edge and half screw dislocations of f.c.c. type, A_i would have to have a rather high value: $A_i \simeq 8\,(2)$, as can be seen in Fig. 2(*a*). If, however, A_i is assumed to have a more realistic, smaller value, not too far from unity, then the $q_{\rm exp}$ value would indicate the presence of mainly screw dislocations. The dislocation structures in C_{60} fullerene crystals have been studied by high-resolution TEM by Muto *et al.* (1993), who found that the observed defects were very similar to those in f.c.c. metals and alloys with low stacking-fault energy. From the present evaluation and the TEM results of Muto *et al.* (1993), we conclude that: (i) A_i is

larger than unity and (ii) the dislocations are most probably f.c.c. type screw dislocations. Furthermore, taking into account that the Rb_3C_{60} fullerite crystals were subjected to X-rays directly after preparation, without extra deformation (Fischer *et al.*, 1995), the dislocations prevailing in the crystals are most probably as-grown dislocations. Since screw dislocations have a smaller self energy per unit length than edge dislocations, the result that mainly screws were found is also energetically reasonable.

9. Conclusions

The dislocation model of the mean square strain enables the rationalization of strain anisotropy in terms of crystallite size and dislocations. A crucial point in the model and procedure is the use of dislocation contrast factors, C. The values of C depend on (i) hkl, (ii) the relative directions of the Burgers and line vectors of dislocations and the diffraction vector, and (iii) the elastic constants of the crystal. In a recent work it was shown that in a polycrystalline specimen or if the different slip systems are populated randomly by dislocations, the C factors can be averaged for one particular

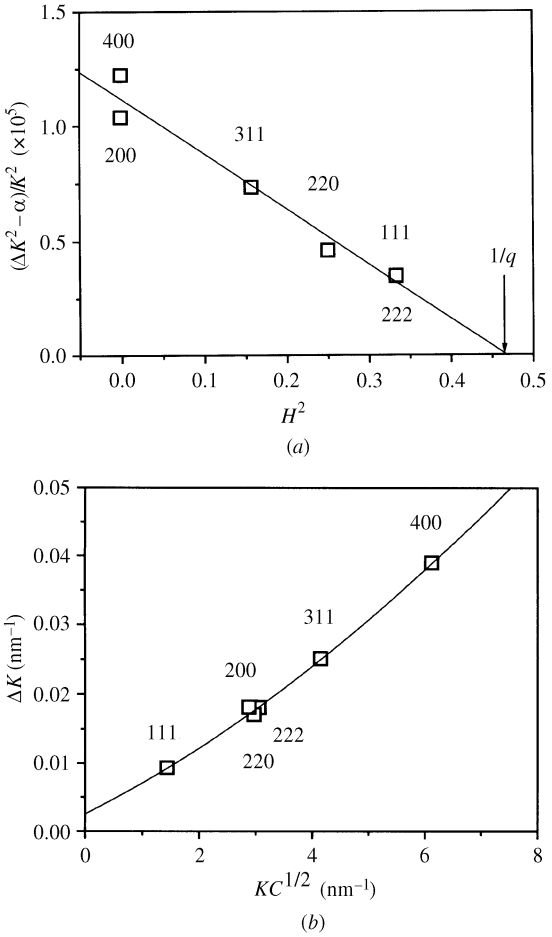

Fig. 3. The FWHM of submicrometre-grain-size copper plotted (*a*) according to equation (12) and (*b*) according to the *modified* Williamson–Hall plot. (The specimen was kindly provided by Professor R. Valiev.)

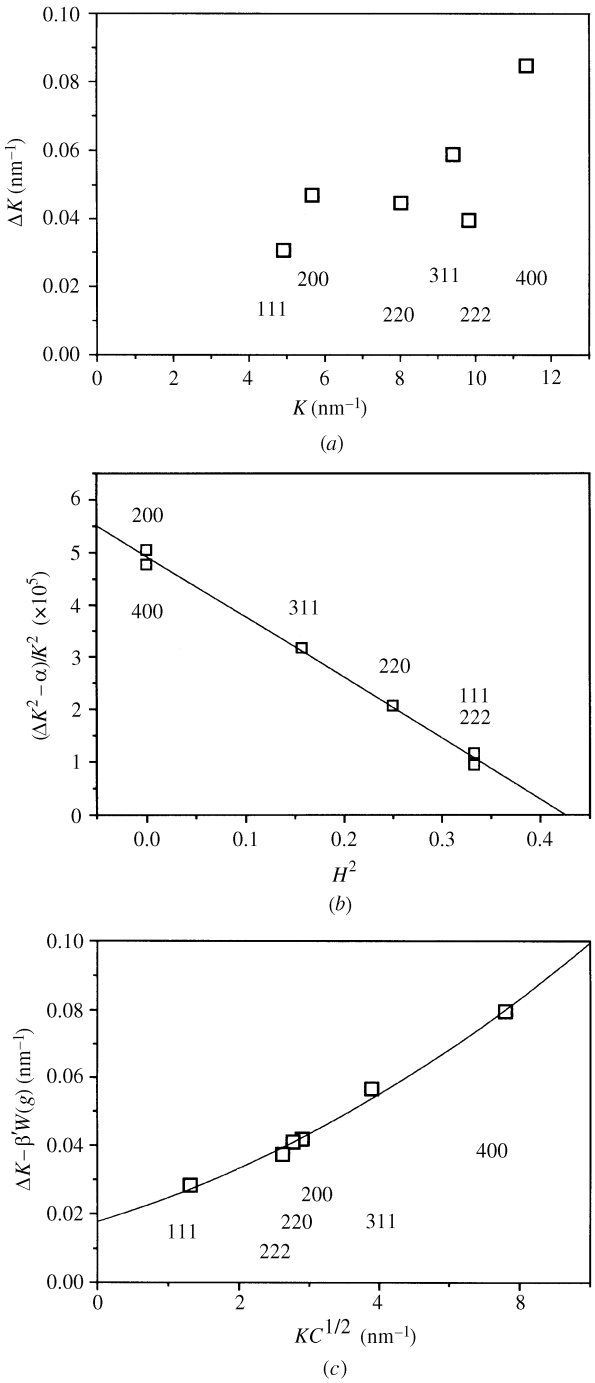

Fig. 4. The FWHM of electrodeposited nanocrystalline nickel (*a*) in the *classical* Williamson–Hall plot, (*b*) according to equation (12) and (*c*) according to the *modified* Williamson–Hall plot.

hkl type. The average contrast factors $\bar{C}$ become a simple function of the elastic constants of the crystal, as given in equation (7): $\bar{C} = \bar{C}_{h00}(1 - qH^2)$. The values of $\bar{C}_{h00}$ and q have been calculated and compiled for f.c.c. and b.c.c. crystals containing dislocations with ⟨110⟩ and ⟨111⟩ Burgers vectors. The compilation was made graphically, presenting the data *versus* the anisotropy factors, A_i, and by giving the $\bar{C}_{h00}(A_i, c_{12}/c_{44})$ and $q(A_i, c_{12}/c_{44})$ functions in parametrized form. The experimental values of q can be discussed in terms of dislocation types and elastic constants. By the dislocation model, the explanation of strain anisotropy has been given a sound physical basis. The whole scheme of the compiled contrast factors provides a tool enabling: (*a*) the rationalization of strain anisotropy in terms of the *modified* Williamson–Hall plot and the *modified* Warren–Averbach method, (*b*) the discussion of strain anisotropy in terms of dislocations, and (*c*) the use of all measured Bragg reflections in the determination of crystallite size. The use of the scheme of contrast factors and the whole procedure has been illustrated by the examples of three different materials: (i) plastically deformed submicrometre-grain-size copper, (ii) electrodeposited nanocrystalline nickel, and (iii) Rb-doped Rb_3C_{60} fullerite.

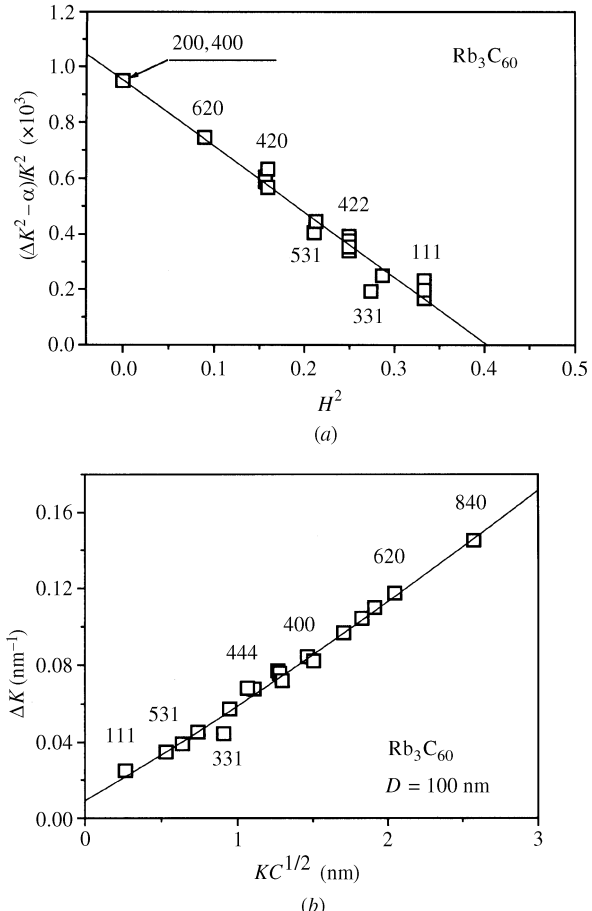

Fig. 5. The FWHM of Rb_3C_{60} plotted (*a*) according to equation (12) and (*b*) according to the *modified* Williamson–Hall plot. [The values of the FWHM were obtained through the courtesy of Fischer *et al.* (1995).]

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